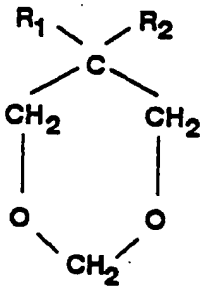




INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(51) International Patent Classification ⁵ : C10M 105/18, 129/20 // C10N 40:36	A1	(11) International Publication Number: WO 94/00539 (43) International Publication Date: 6 January 1994 (06.01.94)
(21) International Application Number: PCT/SE93/00446 (22) International Filing Date: 19 May 1993 (19.05.93) (30) Priority data: 9201978-5 26 June 1992 (26.06.92) SE (71) Applicant (for all designated States except US): PERSTORP AB [SE/SE]; S-284 80 Perstorp (SE). (72) Inventors; and (75) Inventors/Applicants (for US only) : SÖRENSEN, Kent [SE/SE]; Rönnvägen 4, S-284 35 Perstorp (SE). GARPVALL, Lars-Olof [SE/SE]; Södra Kyrkogatan 20-22, S-252 23 Helsingborg (SE). JOHANSSON, Mats [SE/SE]; Uardavägen 52C, S-224 71 Lund (SE).		(74) Agent: STENBERG, Yngve; Perstorp AB, S-284 80 Perstorp (SE). (81) Designated States: AT, AU, BB, BG, BR, CA, CH, CZ, DE, DK, ES, FI, GB, HU, JP, KP, KR, LK, LU, MG, MN, MW, NL, NO, NZ, PL, PT, RO, RU, SD, SE, SK, UA, US, European patent (AT, BE, CH, DE, DK, ES, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, ML, MR, NE, SN, TD, TG). Published <i>With international search report.</i>
(54) Title: THE USE OF 1,3-DIOXANES IN LUBRICANT AND RELEASE AGENTS <div style="text-align: center; margin: 20px 0;">  <div style="position: absolute; left: 630px; top: 690px;">(I)</div> </div>		
(57) Abstract <p>The use of a component based on 1,3-dioxane compounds, especially 1,3-dioxane alcohols, and derivatives thereof in lubricant and release agents. The invention also relates to a lubricant and release agent based on or containing 1,3-dioxanes. The component is of general formula (I) in which formula R₁ or R₂ each independently is hydrogen, hydroxyl, an alkyl moiety, a substituted alkyl moiety, an alkenyl moiety, a substituted alkenyl moiety, a cycloalkyl moiety, a substituted cycloalkyl moiety, a cycloalkenyl moiety, a substituted cycloalkenyl moiety, an aryl moiety, a substituted aryl moiety, an aralkyl moiety, an alkenyl moiety, an aralkenyl moiety and/or an alkenaryl moiety.</p>		

FOR THE PURPOSES OF INFORMATION ONLY

Codes used to identify States party to the PCT on the front pages of pamphlets publishing international applications under the PCT.

AT	Austria	FR	France	MR	Mauritania
AU	Australia	GA	Gabon	MW	Malawi
BB	Barbados	GB	United Kingdom	NE	Niger
BE	Belgium	GN	Guinea	NL	Netherlands
BF	Burkina Faso	GR	Greece	NO	Norway
BG	Bulgaria	HU	Hungary	NZ	New Zealand
BJ	Benin	IE	Ireland	PL	Poland
BR	Brazil	IT	Italy	PT	Portugal
BY	Belarus	JP	Japan	RO	Romania
CA	Canada	KP	Democratic People's Republic of Korea	RU	Russian Federation
CF	Central African Republic	KR	Republic of Korea	SD	Sudan
CG	Congo	KZ	Kazakhstan	SE	Sweden
CH	Switzerland	LI	Liechtenstein	SI	Slovenia
CI	Côte d'Ivoire	LK	Sri Lanka	SK	Slovak Republic
CM	Cameroon	LU	Luxembourg	SN	Senegal
CN	China	LV	Latvia	TD	Chad
CS	Czechoslovakia	MC	Monaco	TG	Togo
CZ	Czech Republic	MG	Madagascar	UA	Ukraine
DE	Germany	ML	Mali	US	United States of America
DK	Denmark	MN	Mongolia	UZ	Uzbekistan
ES	Spain			VN	Viet Nam
FI	Finland				

THE USE OF 1,3-DIOXANES IN LUBRICANT AND RELEASE AGENTS

The present invention relates to the use of a component based on 1,3-dioxane compounds, especially 1,3-dioxane alcohols, and derivatives thereof in lubricant and release agents. This invention also relates to a lubricant and release agent based on or containing 1,3-dioxane compounds as above.

Lubricant and release agents are normally used in connection with metal cutting, tapping, threading, reaming etc. as well as for concrete casting. Further and frequent application areas include utensils and plants for refrigeration, air conditioners, jet and conventional combustion engines, hydraulic fluids and the like.

Above exemplified application areas most often involve lubricant and release agents based on or containing mineral oils and one or more property adjusting additives such as EP-additives (EP = Extreme Pressure). EP-additives are mostly based on chloric, sulphuric and/or phosphatic compounds of the paraffin type.

Products containing mineral oils give rise to oil mist with pendant oil-polluted air and oil coated equipments in and around a working area. Mineral oils and for instance EP-additives are furthermore known to cause skin irritation, eczema and/or allergic reactions. Carcinogenic effects can not be excluded, as most mineral oils contain for example aromatic hydrocarbons of the benzopyrene type, which compounds at high working temperatures most probably form carcinogenic polyaromatics.

Mineral oils as well as chloric, sulphuric and/or phosphatic compounds give also rise to undesirable ecological effects,

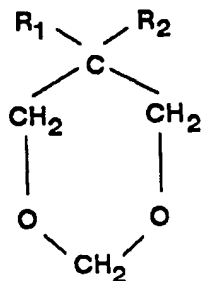
such as gradual concentration of undesired compounds like chlorine, sulphur and/or phosphorous in soil and water. Furthermore, mineral oils are not or only to a very low extent biodegradable, while the component according to the invention per se is biodegradable and based on biodegradable substances.

Mineral oils have per se a limited lubricating and releasing power, why a number of additives must be admixed. Besides property adjusting additives must for instance waterborne emulsions of mineral oils and synthetic lubricants comprise compounds such as emulsifiers and biocides, which compounds may irritate the skin and/or the respiratory passage.

According to the present invention, it has surprisingly been possible to solve above discussed problems by a replacement of mineral oils and/or property adjusting additives with a component based on 1,3-dioxane compounds and/or derivatives thereof.

The lubricating properties have furthermore been improved by utilisation of the component as additive in lubricant and release agents. An addition is performed without any further alteration of the original composition. A suitable addition level is 0.1-20% by weight, preferably 0.5-10% by weight, calculated on included active substances.

The component is of the general formula



in which formula R_1 is $-H$, $-OH$, $-CH_3$, $-C_2H_5$, $-CH_2OR_3$,
 $-CH_2O(C_2H_4O)_nR_3$, $-CH_2O(C_3H_6O)_nR_3$, $-CH_2O(C_4H_8O)_nR_3$,
 $-CH_2O(C_8H_8O)_nR_3$, $-CH_2O(R_5)_m(R_6)_pR_3$ or $-CH_2OR_7$ and R_2 is
 $-H$, $-OH$, $-OR_4$, $-O(C_2H_4O)_nR_4$, $-O(C_3H_6O)_nR_4$, $-O(C_4H_8O)_nR_4$,
 $-O(C_8H_8O)_nR_4$, $-CH_2OR_4$, $-CH_2O(C_2H_4O)_nR_4$, $-CH_2O(C_3H_6O)_nR_4$,
 $-CH_2O(C_4H_8O)_nR_4$, $-CH_2O(C_8H_8O)_nR_4$, $-O(R_5)_m(R_6)_pR_4$,
 $-CH_2O(R_5)_m(R_6)_pR_4$, $-OR_7$ or $-CH_2OR_7$.

R_3 and/or R_4 is each independently selected from any of the below groups:

- (i) hydrogen;
- (ii) an alkyl moiety;
- (iii) a substituted alkyl moiety;
- (iv) an alkenyl moiety;
- (v) a substituted alkenyl moiety;
- (vi) a cycloalkyl moiety;
- (vii) a substituted cycloalkyl moiety;
- (viii) a cycloalkenyl moiety;
- (ix) a substituted cycloalkenyl moiety;
- (x) an aryl moiety;
- (xi) a substituted aryl moiety;
- (xii) an aralkyl moiety, an alkaryl moiety, an aralkenyl moiety and/or an alkenaryl moiety.

R_5 and R_6 are two different substituents of the formula C_xH_yO wherein x is 2, 3, 4 or 8 and y is 4, 6 or 8.

R_7 is defined by the general formula $\begin{array}{c} O \\ || \\ -C-NH-R_8 \end{array}$ in which the substituent R_8 is selected from any of the groups (ii) through (xii).

The mean value \bar{n} for n is 1-60, preferably 1-20, the mean

values \bar{m} for m and \bar{p} for p is 1-59, preferably 1-19 and the sum of the mean values \bar{m} and \bar{p} is 2-60, preferably 2-20.

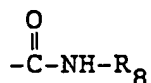
According to one embodiment of the invention, R_1 and/or R_2 is hydroxyl. In such an embodiment of the invention, the used component is a 1,3-dioxane alcohol, such as 1,3-dioxane-5-ol, 5-ethyl-1,3-dioxane-5-methanol, 1,3-dioxane-5,5-dimethanol and/or adducts thereof with for instance ethylene oxide, propylene oxide, butylene oxide and/or styrene oxide. One or more of the carbon atoms in the 1,3-dioxane ring can, furthermore, be methyl, ethyl, butyl, propyl, pentyl, hexyl, heptyl, octyl, nonyl, decyl, undecyl, dodecyl, ethenyl, butenyl, propenyl, pentenyl, hexenyl, heptenyl, octenyl, nonenyl, decenyl, undecenyl and/or dodecenyl substituted.

The component is in above embodiment used as 100% product or in diluted form and is employed as sole constituent or as additive in a lubricant or release agent. The component can as additive suitably comprise 1-80% by weight, preferably 40-80% by weight, calculated on the total formulation.

In a further embodiment, R_3 and R_4 are each independently selected from any of the groups (ii) through (xii) preferably derived from one or more carboxylic acids having 1-6 carboxyl groups and 1-24 carbon atoms, such as abietic acid, acetic acid, behenic acid, benzoic acid, p-tert.butylbenzoic acid, butyric acid, castor fatty acid, dehydrated castor fatty acid, capric acid, caproic acid, caprylic acid, coconut fatty acid, cottonseed fatty acid, crotonic acid, 2-ethylhexanoic acid, formic acid, groundnut fatty acid, heptanoic acid, lauric acid, licanic acid, linolic acid, linolenic acid, montanoic acid, myristic acid, nonanoic acid, isononanoic acid, oleic acid, palmitic acid, propionic acid, ricinoleic acid, soybean fatty acid, stearic acid, isostearic acid, tall oil fatty acid, tallow fatty acid, valeric acid, adipic acid, azelaic

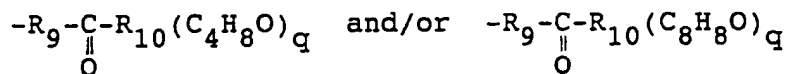
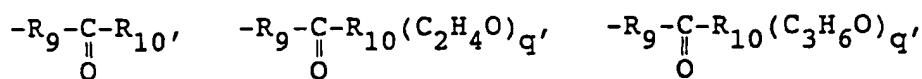
acid, fumaric acid, maleic acid or its anhydride, phthalic acid or its anhydride, isophthalic acid, tetrahydrophthalic acid or its anhydride, hexahydrophthalic acid or its anhydride, sebacic acid, succinic acid or its anhydride, citric acid, trimellitic acid or its anhydride, pyromellitic acid or its dianhydride and/or a mixture of two or more of these acids or anhydrides.

Embodiments in which R_7 is defined by the general formula



the component according to the invention comprises a reaction product and/or a derivative thereof derived from for instance a 1,3-dioxane alcohol as previously exemplified and at least one isocyanate having the general formula $\text{O}=\text{C}=\text{N}-\text{R}_8$. R_8 is in these embodiments preferably defined by having 4-48, most preferably 8-24 carbon atoms.

In yet a further embodiment R_3 and/or R_4 is each independently a group of the formula



wherein the mean value \bar{q} for q is 1-60, preferably 1-20 and R_9 is selected from any of the groups (ii) through (xii) preferably derived from one or more carboxylic acids having 2-6 carboxyl groups and 4-10 carbon atoms, such as adipic acid, azelaic acid, fumaric acid, maleic acid or its anhydride, phthalic acid or its anhydride, isophthalic acid, tetrahydrophthalic acid or its anhydride, hexahydrophthalic

acid or its anhydride, sebacic acid, succinic acid or its anhydride, citric acid, trimellitic acid or its anhydride, pyromellitic acid or its dianhydride and/or a mixture of two or more of these acids and R_{10} is hydroxyl or selected from any of the groups (ii) through (xii) derived from one or more alcohols having 1-8 hydroxyl groups and 1-24 carbon atoms, such as methanol, ethanol, butanol, isobutanol, propanol, isopropanol, pentanol, hexanol, octanol, 2-ethylhexanol, ethoxyethanol, cetyl alcohol, trimethylolpropane diallyl ether, pentaerythritol triallyl ether, glycerol diallyl ether, 1,3-dioxane-5-ol, cyclohexane-dimethanol, 5-ethyl-1,3-dioxane-5-methanol, 1,3-butanediol, 1,4-butanediol, 1,6-hexanediol, pentanediol, neopentyl glycol, hexylene glycol, 2-methyl-1,3-propanediol, 2-methyl-2-ethyl-1,3-propanediol, 2-methyl-2-butyl-1,3-propanediol, 2-ethyl-2-butyl-1,3-propanediol, trimethylpentanediol, trimethylolpropane monoallyl ether, pentaerythritol diallyl ether, glycerol monoallyl ether, 1,3-dioxane-5,5-dimethanol, glycerol, trimethylolethane, trimethylolpropane, pentaerythritol monoallyl ether, pentaerythritol, ditrimethylolpropane, dipentaerythritol, tripentaerythritol, sorbitol and/or a mixture of two or more of these alcohols.

In alternative embodiments, R_3 , R_4 , R_9 and/or R_{10} is each independently selected from any of the groups (ii) through (xii) preferably derived from one or more hydroxycarboxylic acids having 1-3 carboxyl groups, 1-4 hydroxyl groups and 2-24 carbon atoms, such as dimethylolpropionic acid and hydroxypivalic acid. R_3 and/or R_4 can also be selected from group (xii) wherein the alkyl moiety derived from one or more glycidyl esters of monofunctional carboxylic acids having 1-24 carbon atoms or each independently be selected from any of the groups (ii) through (xii) derived from one or more α -epoxides having 5-24 carbon atoms.

In embodiments of the component holding one or more unreacted carboxyl groups these groups can, in order to obtain a water dilutability, be neutralised with a suitable basic compound such as ammonia, an amine, a hydroxide or the like. Ammonia or an amine such as monoethanol amine, diethanol amine, triethanol amine, N,N-dimethylethanol amine, N,N-dimethylaminomethylpropanol, aminomethylpropanol, triethyl amine and/or morpholine are preferred neutralising agents.

Advantages obtained by the present invention include improved working conditions, which conditions are improved through a replacement of mineral oils and/or additives, whereby the skin irritating, allergic and/or eczema producing properties of these compounds are eliminated or reduced. Furthermore, problems caused by oil mist and oil coating can be avoided or reduced by selecting the substituents $R_1 - R_{10}$ in such a way within the scope of the claims that a reduced volatility, compared to normally used mineral oils, is obtained. The ecological disadvantages of using mineral oils and for instance chloric, sulphuric and/or phosphatic additives and the like are, due to the biodegradability of the component according to the invention as well as due to the fact that the component does not contain chloric, sulphuric and/or phosphatic substances, avoided.

Further advantages obtained by using the component according to the invention include improved and excellent lubricating power, excellent thermal stability and excellent solubility in most organic media.

The component according to the present invention can suitably be utilised as additive to compositions as disclosed in the American patent 4,405,471 and the European patent application 89 913 158.5 or replace components included in said compositions. Esters forming part of a lubricating fluid as described in for instance Examples 8-14 of above American patent and

Examples 8-12 of above European patent application can be combined with or wholly and/or partly replaced by esters prepared from one or more 1,3-dioxane alcohols and/or derivatives thereof such as alkoxylated 1,3-dioxane alcohols and one or more suitable acids, as previously disclosed, which esters possibly have been neutralised with a suitable amine or the like.

The invention will be further explained in connection with enclosed examples in which some preferred embodiments of the invention are disclosed as follows:

Example 1: Preparation of 5-ethyl-1,3-dioxane-5-methanol used as final or intermediate product according to the invention.

Examples 2 and 3: Preparations of monoesters of 5-ethyl-1,3-dioxane-5-methanol used as final or intermediate product according to the invention.

Examples 4-6: Evaluations of lubricant agents, cutting fluids, containing esters prepared according to Examples 2 and 3.

Examples 7 and 8: Evaluations of the lubricating power of the products obtained according to Examples 1 and 2.

Example 9: Evaluation of release agents in concrete casting. The release agents are based on products obtained according to Examples 1 and 3 and are compared with a commercial release agent.

The present invention is not limited to disclosed embodiments. The component according to invention as well as its properties

can be varied within the scope of the claims by selecting the substituents $R_1 - R_{10}$ to comply with particular requirements.

EXAMPLE 1

3.0 moles of trimethylolpropane, 3.6 moles of paraformaldehyde (94%) and 0.40 g of paratoluene sulphonic acid were charged and mixed in a 4-necked reaction flask equipped with a nitrogen inlet, a stirrer and a cooler provided with a water-trap (Dean-Stark). The mixture was under stirring heated to 130°C and kept at this temperature for 60 minutes. Obtained reaction product was neutralised with powdered sodium hydroxide and thereafter vacuum distilled using a short Vigreux column, which distillation resulted in the following fractions:

	<u>Boiling point</u>	<u>Pressure</u>	<u>Amount</u>
Fraction I:	100°C	2 mm Hg	90 g
Fraction II:	100-105°C	2 mm Hg	168 g
Fraction III:	105-150°C	1 mm Hg	8 g
Residue:	----	----	171 g

Fraction I and II, colourless or slightly yellowish liquids, were mixed and analytically determined to be 5-ethyl-1,3-dioxane-5-methanol having the following properties:

Content:	> 98%
Moisture content:	< 0.05%
Ash content:	< 0.5%
Viscosity at 23°C:	≈ 80 mPas
Density at 23°C:	≈ 1.09 g/cm ³

EXAMPLE 2

1.70 mole of 5-ethyl-1,3-dioxane-5-methanol (obtained in Example 1), 1.615 mole of oleic acid (Edinor TiO5, Henkel KGaA, Fed. Rep. of Germany), 3.5 g of trisnonylphenyl phosphite (antioxidant) and 21 g of xylene (azeotropic solvent) were charged and mixed in a 4-necked reaction flask equipped with a nitrogen inlet, a stirrer and a cooler provided with a water-trap (Dean-Stark). The temperature was raised to 120°C, whereupon 1.4 g of zinc powder (esterification catalyst) was added. The temperature of the reaction mixture was now raised to 230°C and maintained until an acid value of less than 2 mg KOH/g was obtained, whereupon the remaining xylene was evaporated at a vacuum of 15 mm Hg. The resulting product was cooled to room temperature, a filter aid (Celite) was added in an amount of 2% and the product was filtered to remove organozinc compounds.

Obtained monoester of 5-ethyl-1,3-dioxane-5-methanol and oleic acid, exhibited the following properties:

Acid value:	1.9 mg KOH/g
Hydroxyl value:	7 mg KOH/g
Viscosity at 23°C:	47 mPas
Colour value:	4-5 Gardner

EXAMPLE 3

2.40 moles of 5-ethyl-1,3-dioxane-5-methanol (obtained in Example 1), 2.28 moles of caprylic-capric acid (C_8-C_{10} acids, Karlshamns AB, Sweden), 3.5 g of trisnonylphenyl phosphite (antioxidant), 21 g of xylene (azeotropic solvent) and 0.7 g of an esterification catalyst (Fascat 4100, M&T Chemicals B.V., The Netherlands) were charged and mixed in a 4-necked

reaction flask equipped with a nitrogen inlet, a stirrer and a cooler provided with a water-trap (Dean-Stark). The temperature of the reaction mixture was raised to 230°C and maintained until an acid value of less than 0.5 mg KOH/g was obtained. The remaining xylene was thereafter evaporated at a vacuum of 15 mm Hg and a temperature of 180°C and the resulting product was cooled to room temperature.

Obtained monoester of 5-ethyl-1,3-dioxane-5-methanol and caprylic-capric acid, exhibited the following properties:

Acid value:	0.3 mg KOH/g
Hydroxyl value:	13 mg KOH/g
Viscosity at 23°C:	31 mPas
Colour value:	1-2 Gardner

EXAMPLE 4

1.5% by weight of 5-ethyl-1,3-dioxane-5-methanol (obtained in Example 1) was added to a commercially available mineral oil based cutting fluid (Peralub 6000, Perstorp AB, Sweden) having a mineral oil content of 23% by weight. The two cutting fluids, the commercially available (Sample 1) and the 5-ethyl-1,3-dioxane-5-methanol modified (Sample 2), were diluted with water to an utility concentration of 5% by weight.

The two cutting fluids were evaluated by tapping in aluminium. The amount of aluminium adhering to the tap were visually determined and the result was used as a basis for grading the lubricating properties.

A grading of 1-5 was used, wherein:

1 = Poor lubricating properties - A large amount of aluminium adheres to the tap.

5 = Excellent lubricating properties - A very small amount or no aluminium adheres to the tap.

The following results were obtained:

	<u>Grading</u>
Sample 1:	3
Sample 2:	5

Above grading indicates that a small addition of 5-ethyl-1,3-dioxane-5-methanol, without any alteration of other included constituents, results in substantially increased lubricating properties.

EXAMPLE 5

Example 4 was repeated with the difference that 5-ethyl-1,3-dioxane-5-methanol was replaced by the monoester obtained in Example 2.

Sample 1 = Commercially available cutting fluid.

Sample 2 = Monoester modified cutting fluid.

The following results were obtained:

	<u>Grading</u>
Sample 1:	3
Sample 2:	4

Above grading indicates that a small addition of the mono-ester of oleic acid and 5-ethyl-1,3-dioxane-5-methanol, without any alteration of other included constituents, results in increased lubricating properties.

EXAMPLE 6

Example 4 was repeated with the difference that 5-ethyl-1,3-dioxane-5-methanol was replaced by the monoester obtained in Example 3.

Sample 1 = Commercially available cutting fluid.

Sample 2 = Monoester modified cutting fluid.

The following results were obtained:

	<u>Grading</u>
Sample 1:	3
Sample 2:	4

Above grading indicates that a small addition of the monoester of caprylic-capric acid and 5-ethyl-1,3-dioxane-5-methanol, without any alteration of other included constituents, results in increased lubricating properties.

EXAMPLE 7

1.5% by weight of 5-ethyl-1,3-dioxane-5-methanol (obtained in Example 1) was added to a commercially available mineral oil (Nyflex 810, Nynäs Petroleum AB, Sweden). The specific surface pressure was, for the two samples, determined according to the German V K I S Arbeitsblatt 6 of June 1975. A high specific surface pressure indicates a high lubricating

power with pendant reduced abrasion of equipment such as taps, drills, cutters and the like.

Sample 1: Commercially available mineral oil.

Sample 2: 5-ethyl-1,3-dioxane-5-methanol modified mineral oil.

The following results were obtained:

Specific Surface Pressure

Sample 1: 10 N/mm²

Sample 2: 20 N/mm²

Above results give at hand that a small addition of 5-ethyl-1,3-dioxane-5-methanol to a mineral oil highly increases its lubricating power.

EXAMPLE 8

Example 7 was repeated with the difference that 5-ethyl-1,3-dioxane-5-methanol was replaced by the monoester obtained in Example 2.

Sample 1: Commercially available mineral oil.

Sample 2: Monoester modified mineral oil.

The following results were obtained:

Specific Surface Pressure

Sample 1: 10 N/mm²

Sample 2: 25 N/mm²

Above results give at hand that a small addition of the mono-ester of oleic acid and 5-ethyl-1,3-dioxane-5-methanol to a mineral oil highly increases its lubricating power.

EXAMPLE 9

A commercially available release agent (Lasol M100, Byggekemi i Nol AB, Sweden) based on mineral oil was evaluated in comparison with two embodiments of the invention

Sample 1: A mixture of 40 g of 5-ethyl-1,3-dioxane-5-methanol (obtained in Example 1) and 60 g of water.

and

Sample 2: The monoester (obtained in Example 3) of caprylic-capric acid and 5-ethyl-1,3-dioxane-5-methanol

according to the following method:

- A release agent is sprayed onto the inner side of a cubic steel cast, in which a cube of ordinary concrete, containing Standard Portland cement, is casted. The cube is after 24 hours released from the cast and a visual inspection with regard to adhering concrete is performed on the interior of the cast. The concrete cube is visually inspected with regard to surface roughness, hardness and blistering.

The amount of adhering concrete and the appearance of the surface of the concrete cube are bases for grading the release agent. The surface should be free from blisters, even and hard, while the interior of the cast should be free from adhering concrete.

A grading of 1-3 is used, wherein:

1 = Not approved

3 = Approved

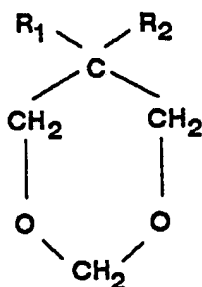
The following results were obtained:

	<u>Grading</u>
Commercial release agent:	3
Sample 1:	3
Sample 2:	3

Above results show that the environmentally more suitable component according to the invention, can replace a mineral oil based release agent without deterioration of the technical properties.

CLAIMS

1. Use of a 1,3-dioxane compound of the general formula



in which formula

R₁ is -H, -OH, -CH₃, -C₂H₅, -CH₂OR₃, -CH₂O(C₂H₄O)_nR₃,
 -CH₂O(C₃H₆O)_nR₃, -CH₂O(C₄H₈O)_nR₃, -CH₂O(C₈H₈O)_nR₃,
 -CH₂O(R₅)_m(R₆)_pR₃ or -CH₂OR₇;

and

R₂ is -H, -OH, -OR₄, -O(C₂H₄O)_nR₄, -O(C₃H₆O)_nR₄,
 -O(C₄H₈O)_nR₄, -O(C₈H₈O)_nR₄, -CH₂OR₄, -CH₂O(C₂H₄O)_nR₄,
 -CH₂O(C₃H₆O)_nR₄, -CH₂O(C₄H₈O)_nR₄, -CH₂O(C₈H₈O)_nR₄,
 -O(R₅)_m(R₆)_pR₄, -CH₂O(R₅)_m(R₆)_pR₄, -OR₇ or -CH₂OR₇;

wherein

R₃ and/or R₄ each independently is selected from any of the groups:

- (i) hydrogen;
- (ii) an alkyl moiety;
- (iii) a substituted alkyl moiety;
- (iv) an alkenyl moiety;
- (v) a substituted alkenyl moiety;

- (vi) a cycloalkyl moiety;
- (vii) a substituted cycloalkyl moiety;
- (viii) a cycloalkenyl moiety;
- (ix) a substituted cycloalkenyl moiety;
- (x) an aryl moiety;
- (xi) a substituted aryl moiety;
- (xii) an aralkyl moiety, an alkaryl moiety, an aralkenyl moiety and/or an alkenaryl moiety;

and wherein

R_5 and R_6 are two different substituents of the formula C_xH_yO wherein x is 2, 3, 4 or 8 and y is 4, 6 or 8;

and wherein

R_7 is defined by the general formula $\overset{\text{O}}{\parallel}\text{-C-NH-R}_8$ in which the substituent R_8 is selected from any of the groups (ii) through (xii);

and wherein

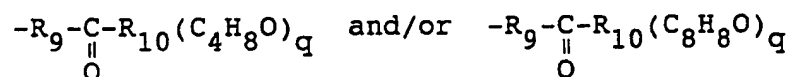
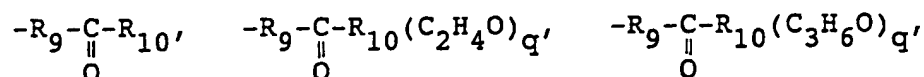
the mean value \bar{n} for n is 1-60, preferably 1-20, the mean value \bar{m} for m is 1-59, preferably 1-19, the mean value \bar{p} for p is 1-59, preferably 1-19 and the sum of the mean values \bar{m} and \bar{p} is 2-60, preferably 2-20;

as a component in lubricant and release agents.

2. Use according to claim 1 characterised in, that one or more of the carbon atoms in the 1,3-dioxane ring are methyl, ethyl, butyl, propyl, pentyl, hexyl, heptyl, octyl, nonyl, decyl, undecyl, dodecyl, ethenyl, butenyl, propenyl, pentenyl, hexenyl, heptenyl, octenyl,

nonenyl, decenyl, undecenyl and/or dodecenyl substituted

3. Use according to claim 1 or 2 characterised in, that R_3 and/or R_4 each independently is selected from any of the groups (ii) through (xii) derived from one or more carboxylic acids having 1-6 carboxyl groups 1-24 carbon atoms.
4. Use according to claim 1 or 2 characterised in, that R_3 and/or R_4 each independently is a group of the formula



wherein

R_9 is selected from any of the groups (ii) through (xii) derived from one or more carboxylic acids having 2-6 carboxyl groups and 4-10 carbon atoms;

and wherein

R_{10} is hydroxyl or is selected from any of the groups (ii) through (xii) derived from one or more alcohols having 1-8 hydroxyl groups and 1-24 carbon atoms;

and wherein

the mean value \bar{q} for q is 1-60, preferably 1-20.

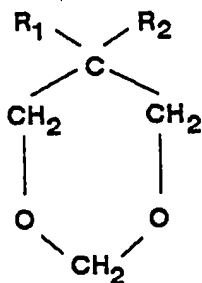
5. Use according to claim 1 or 2 characterised in, that R_3 and/or R_4 each independently is selected from group (xii) wherein the alkyl moiety is derived from one or more glycidyl esters of monofunctional carboxylic acids having 1-24 carbon atoms.
6. Use according to claim 1 or 2 characterised in, that R_3 and/or R_4 each independently is selected from any of the groups (ii) through (xii) derived from one or more α -epoxides having 5-24 carbon atoms.
7. Use according to claim 1 or 2 characterised in, that R_3 , R_4 , R_9 and/or R_{10} each independently is selected from any of the groups (ii) through (xii) derived from one or more hydroxycarboxylic acids having 1-3 carboxyl groups, 1-4 hydroxyl groups and 2-24 carbon atoms.
8. Use according to claim 3 characterised in, that R_3 and/or R_4 each independently is a moiety derived from abietic acid, acetic acid, behenic acid, benzoic acid, p-tert.butylbenzoic acid, butyric acid, castor fatty acid, dehydrated castor fatty acid, capric acid, caproic acid, caprylic acid, coconut fatty acid, cottonseed fatty acid, crotonic acid, 2-ethylhexanoic acid, formic acid, groundnut fatty acid, heptanoic acid, lauric acid, licanic acid, linoic acid, linolenic acid, montanoic acid, myristic acid, nonanoic acid, isononanoic acid, oleic acid, palmitic acid, propionic acid, ricinoleic acid, soybean fatty acid, stearic acid, isostearic acid, tall oil fatty acid, tallow fatty acid, valeric acid, adipic acid, azelaic acid, fumaric acid, maleic acid or its anhydride, phthalic acid or its anhydride, isophthalic acid, tetrahydrophthalic acid

or its anhydride, hexahydrophthalic acid or its anhydride, sebacic acid, succinic acid or its anhydride, citric acid, trimellitic acid or its anhydride, pyromellitic acid or its dianhydride and/or a mixture of two or more of these acids.

9. Use according to claim 1 or 2 characterised in, that R_8 is defined by having 4-48, preferably, 8-24 carbon atoms.
10. Use according to claim 4 characterised in, that R_9 is a moiety derived from adipic acid, azelaic acid, fumaric acid, maleic acid or its anhydride, phthalic acid or its anhydride, isophthalic acid, tetrahydrophthalic acid or its anhydride, hexahydrophthalic acid or its anhydride, sebacic acid, succinic acid or its anhydride, citric acid, trimellitic acid or its anhydride, pyromellitic acid or its dianhydride and/or a mixture of two or more of these acids.
11. Use according to claim 4 characterised in, that R_{10} is a moiety derived from methanol, ethanol, butanol, isobutanol, propanol, isopropanol, pentanol, hexanol, octanol, 2-ethylhexanol, ethoxyethanol, cetyl alcohol, trimethylolpropane diallyl ether, pentaerythritol triallyl ether, glycerol diallyl ether, 1,3-dioxane-5-ol, cyclohexanedimethanol, 5-ethyl-1,3-dioxane-5-methanol, 1,3-butanediol, 1,4-butanediol, 1,6-hexanediol, pentanediol, neopentyl glycol, hexylene glycol, 2-methyl-1,3-propanediol, 2-methyl-2-ethyl-1,3-propanediol, 2-methyl-2-butyl-1,3-propanediol, 2-ethyl-2-butyl-1,3-propanediol, trimethylpentanediol, trimethylolpropane monoallyl ether,

pentaerythritol diallyl ether, glycerol monoallyl ether, 1,3-dioxane-5,5-dimethanol, glycerol, trimethylolethane, trimethylolpropane, pentaerythritol monoallyl ether, pentaerythritol, ditrimethylolpropane, dipentaerythritol, tripentaerythritol, sorbitol and/or a mixture of two or more of these alcohols.

12. Use according to any of the claims 1, 2, 3, 5, 7 or 8 characterised in, that unreacted carboxyl groups are neutralised with at least one basic compound, preferably ammonia or an amine such as monoethanol amine, diethanol amine, triethanol amine, N,N-dimethylethanol amine, N,N-dimethylaminomethylpropanol, triethyl amine aminomethylpropanol and/or morpholine.
13. Lubricant and release agent comprising at least one component composed of at least one 1,3-dioxane compound of the general formula



in which formula

R_1 is $-H$, $-OH$, $-CH_3$, $-C_2H_5$, $-CH_2OR_3$, $-CH_2O(C_2H_4O)_nR_3$, $-CH_2O(C_3H_6O)_nR_3$, $-CH_2O(C_4H_8O)_nR_3$, $-CH_2O(C_8H_8O)_nR_3$, $-CH_2O(R_5)_m(R_6)_pR_3$ or $-CH_2OR_7$;

and

R_2 is $-H$, $-OH$, $-OR_4$, $-O(C_2H_4O)_nR_4$, $-O(C_3H_6O)_nR_4$,
 $-O(C_4H_8O)_nR_4$, $-O(C_8H_8O)_nR_4$, $-CH_2OR_4$, $-CH_2O(C_2H_4O)_nR_4$,
 $-CH_2O(C_3H_6O)_nR_4$, $-CH_2O(C_4H_8O)_nR_4$, $-CH_2O(C_8H_8O)_nR_4$,
 $-O(R_5)_m(R_6)_pR_4$, $-CH_2O(R_5)_m(R_6)_pR_4$, $-OR_7$ or $-CH_2OR_7$;

wherein

R_3 and/or R_4 each independently is selected from any of the groups

- (i) hydrogen;
- (ii) an alkyl moiety;
- (iii) a substituted alkyl moiety;
- (iv) an alkenyl moiety;
- (v) a substituted alkenyl moiety;
- (vi) a cycloalkyl moiety;
- (vii) a substituted cycloalkyl moiety;
- (viii) a cycloalkenyl moiety;
- (ix) a substituted cycloalkenyl moiety;
- (x) an aryl moiety;
- (xi) a substituted aryl moiety;
- (xii) an aralkyl moiety, an alkaryl moiety, an aralkenyl moiety and/or an alkenaryl moiety;

and wherein

R_5 and R_6 are two different substituents of the formula C_xH_yO wherein x is 2, 3, 4 or 8 and y is 4, 6 or 8;

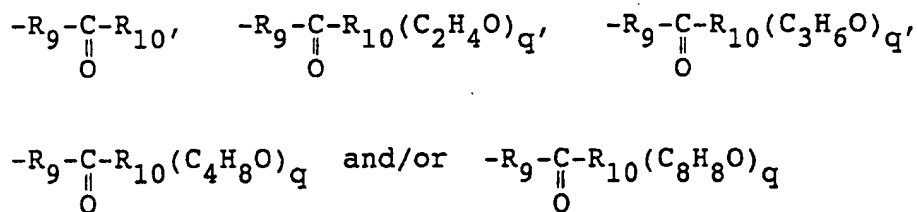
and wherein

R_7 is defined by the general formula $\overset{O}{\parallel}-C-NH-R_8$ in which the substituent R_8 is selected from any of the groups (ii) through (xii);

and wherein

the mean value \bar{n} for n is 1-60, preferably 1-20, the mean value \bar{m} for m is 1-59, preferably 1-19, the mean value \bar{p} for p is 1-59, preferably 1-19 and the sum of the mean values \bar{m} and \bar{p} is 2-60, preferably 2-20.

14. Lubricant and release agent according to claim 13 characterised in, that one or more of the carbon atoms in the 1,3-dioxane ring of the component are methyl, ethyl, butyl, propyl, pentyl, hexyl, heptyl, octyl, nonyl, decyl, undecyl, dodecyl, ethenyl, butenyl, propenyl, pentenyl, hexenyl, heptenyl, octenyl, nonenyl, decenyl, undecenyl and/or dodecenyl substituted.
15. Lubricant and release agent according to claim 13 or 14 characterised in, that R_3 and/or R_4 each independently is a group of the formula



wherein

R_9 is selected from any of the groups (ii) through (xii) derived from one or more carboxylic acids having 2-6 carboxyl groups and 4-10 carbon atoms;

and wherein

R_{10} is hydroxyl or is selected from any of the groups (ii) through (xii) derived from one or more alcohols having 1-8 hydroxyl groups and 1-24 carbon atoms;

and wherein

the mean value \bar{q} for q is 1-60, preferably 1-20.

1

INTERNATIONAL SEARCH REPORT

International application No.

PCT/SE 93/00446

A. CLASSIFICATION OF SUBJECT MATTER

IPC5: C10M 105/18, C10M 129/20 // C10N 40:36

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC5: C10M

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

SE,DK,FI,NO classes as above

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	WO, A1, 8805071 (THE LUBRIZOL CORPORATION), 14 July 1988 (14.07.88) -----	1

☐ Further documents are listed in the continuation of Box C. ☒ See patent family annex.

- * Special categories of cited documents:
- "A" document defining the general state of the art which is not considered to be of particular relevance
- "E" earlier document but published on or after the international filing date
- "L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)
- "O" document referring to an oral disclosure, use, exhibition or other means
- "P" document published prior to the international filing date but later than the priority date claimed
- "T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention
- "X" document of particular relevance: the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone
- "Y" document of particular relevance: the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art
- "&" document member of the same patent family

Date of the actual completion of the international search

22 Sept 1993

Date of mailing of the international search report

24 -09- 1993

Name and mailing address of the ISA/
Swedish Patent Office
Box 5055, S-102 42 STOCKHOLM
Facsimile No. +46 8 666 02 86

Authorized officer

Jack Hedlund
Telephone No. +46 8 782 25 00

26/08/93

PCT/SE 93/00446

Form PCT/ISA/210 (patent family annex) (July 1992)